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Study of Optical Property of Gel Grown Mercuric Iodate Crystals

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Abstract. *Mercuric Iodate Crystals were grown by a simple gel technique using diffusion method. The optimum growth conditions were established for the growth of these crystals by changing various parameters such as pH of the gel solution, gel concentration, gel setting time, concentrations of reactants etc. The grown Mercuric Iodate crystals were spherical in shape. These crystals were opaque. The crystals were characterized using UV-VIS Specrophotometrry.*
.***Keywords.*** *Gel technique, mercuric iodate crystals, UV-VIS Specrophotometrry.*

I. Introduction:

A variety of crystals required for the purpose of research and application can be grown in silica gels. The gel medium prevents turbulence and being chemically inert, it provides a three-dimensional crucible which permits the reagents to diffuse at a desirable controlled rate. Its softness and uniform nature of constraining forces that it exerts upon the growing crystals encourages orderly growth.

The growth of single crystals in gel at an ambient temperature, which are sparingly soluble in water, is a fascinating alternative to the techniques involving high temperature and expensive equipments. During the last few years, successful application of gel growth technique has been demonstrated by the preparation of single crystals of alkaline earth metal iodate. The gel growth technique appeared quite attractive for growing crystals of such compounds on account of its unique advantages in terms of crystals produced and the simplicity of process.

In the present work, crystals of mercuric iodate were grown by gel technique using diffusion method. Optimum growth conditions for crystals were determined. Optimum conditions were established by varying various parameters.

II. Materials and Method

Test tubes were used as crystallizing vessels. The silica gel was used as a growth media. Gel was prepared from aqueous solution of sodium meta silicate. The gel was acidified by acetic acid. The chemicals used for growth of single crystals of mercuric iodate were CH_3COOH; $Na_2SiO_3.5H_2O$; KIO_3, $NaIO_3$; $Hg(NO_3)_2$. All chemicals were of AR grade.

A series of experiments using different pH values for the gel and the different concentrations for reactants were carried out. Different molar masses were tried to determine the optimum growth conditions. Out of the two reactants, $Hg(NO_3)_2$ was incorporated in gel and KIO_3 was used as the supernatant over the set gel. These experiments yield spherulitic crystals of $Hg(IO_3)_2$ of few mm size. Experiments were also carried out by interchanging the position of reactants. These experiments do not yield any crystals at all. $Hg(NO_3)_2$ having different concentrations was incorporated into the gel. This solution was then transferred to borosil glass tube of diameter 2.5 cm and 25 cm in height. The mouth of the tube was covered by cotton plug. After setting of the gel, it was left for aging for different periods of time. Another reactant having different concentrations was then added as supernatant over the set gel. Experiments were carried out by changing different concentrations of the reactants.

The chemical reaction inside the gel can be expressed as

$$X(NO_3)_2 + 2YIO_3 \rightarrow X(IO_3)_2 + 2YNO_3$$

Where X = Hg and Y = K or Na

III. Results And Discussions

Spherulite shaped crystals of 2 to 6 mm size were obtained. Study of kinetics of growth parameters reveals some interesting information. Spherulite shape of crystals is reported and has been explained previously. The shapes may be explained via a model of sheet of paper crumpled in to folds, the edges being compressed towards the centre. The result of this is a rounded crystal, but at first the basal planes (the plane of the sheet of the paper) are bent at random. These serves as the base for two-dimensional nuclei, which grow and fill up the recesses on the outer surface, thus giving crystal a spherical shape.

Higher density gel sets more rapidly. It decreases nucleation density. Increased density reduces diffusivity of ions which in turn reduces growth rate. Lower density gel takes long time to set and can be easily fractured. Increase in aging of gel reduces number of nucleation centres and growth rate. The reason may be the formation of additional cross-linkages between siloxane chains with increasing gel age, resulting in a gradually reducing cell size. This, in turn, reduces nucleation centres, since many nuclei find themselves in cells of very small size, where further growth is not possible. Insufficient gel aging leads to the formation of fragile gel and often breaks at the time of addition of supernatant. The effect of pH on growth rate was studied by changing pH without a change of gel composition and concentration of reactants. With pH values less than 4, gel takes longer time to set and is unstable. There is no considerable effect on the quality of crystal. Higher pH value gel sets early, becomes turbid, and the size of crystal becomes smaller. As the pH increases, the gel structure changes from distinctly box-like network to a structure of loosely bound platelets, which appear to lack cross-linkages and the cellular nature becomes less distinct.

Less concentration of reactants does not yield any crystals at all. High concentration yields crystals of smaller size with increased nucleation centres. Reported concentration of reactants when used yields smaller spherulites near the gel interface with more number of nuclei. This may be due to high diffusion gradient near the gel interface. As the distance from the gel interface increases, number of nuclei reduces and size of spherulite increases due to smaller concentration gradient. Slow diffusion should lead to better nuclei, which because of their higher energy content should be less likely to reach their critical size.

UV-VIS Specrophotometrry:

Optical property of Mercuric iodate crystals can be studied using UV-VIS spectrophotometer. A fine powdered form of crystals was used as sample. The reflection and absorption spectra of Mercuric iodate crystals have been recorded over the wavelength range 200 to 700 nm using a UV-2450 spectrophotometer of SHIMADZU Scientific instruments at the room temperature. The experiment was carried out in the research laboratory of the physics department at pratap college, Amalner. With the help of this, absorption and reflection spectra are directly obtained through the computer using OOI base 32 software.

The absorption and reflection spectra of Mercuric iodate crystals recorded by UV-2450 spectrophotometer are as shown in the figures 1(a) and 1(b) respectively.

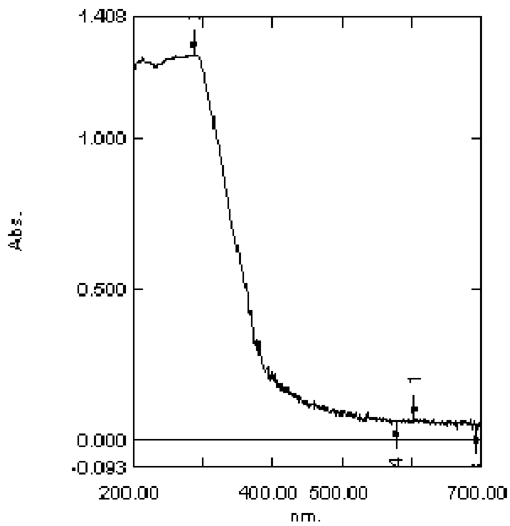

Figure.1(a) Absorption spectra

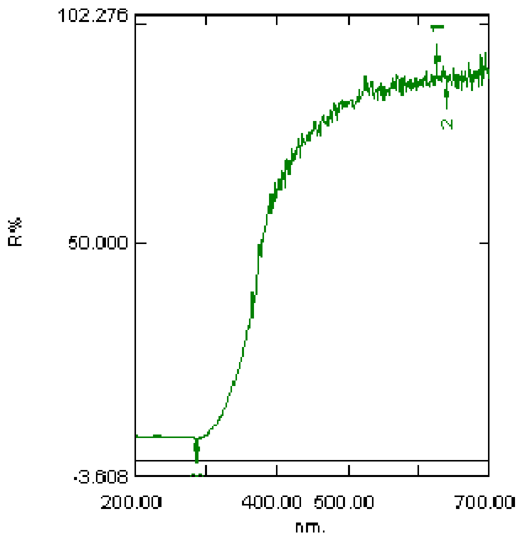

Figure.1(b) Reflection spectra

From the fig.1(a), it is observed that the absorption coefficient is high at lower wavelength and decreases sharply below a certain wavelength for Mercuric iodate crystals.

The excel data recorded by UV-2450 spectrophotometer is as shown in the table 1.

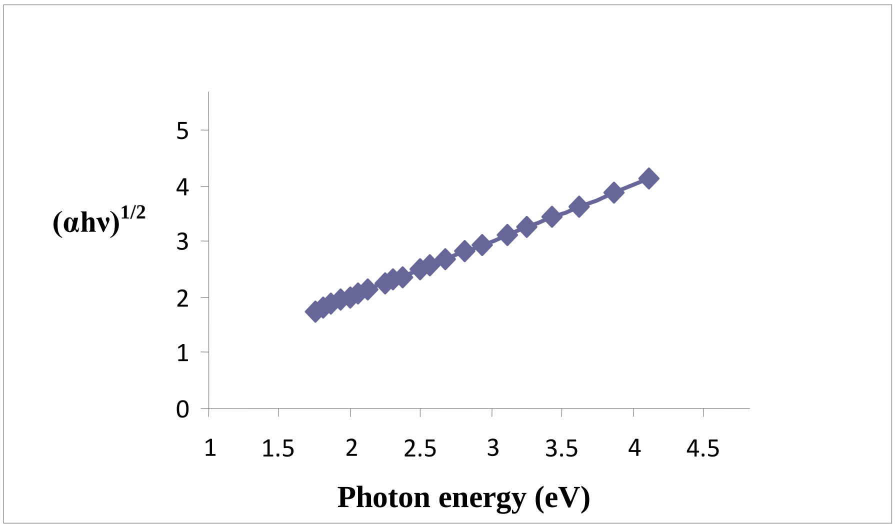

Figure 2(a) The graph of (hν) against $(\alpha h\nu)^{1/2}$

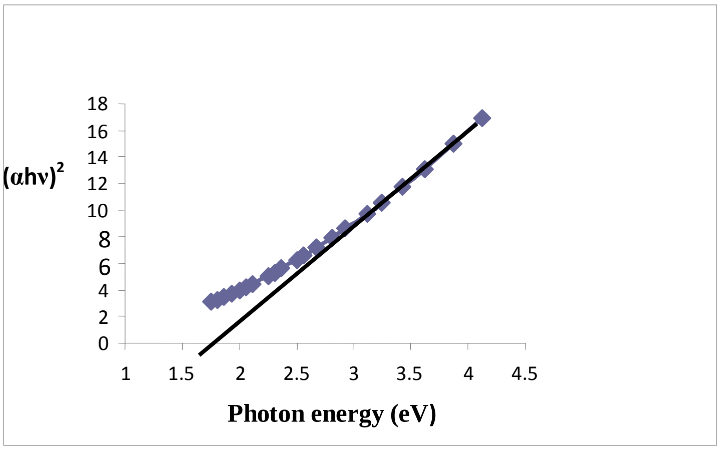

Figure 2(b) The graph of (hν) against $(\alpha h\nu)^2$

Table 1 : Excel data of mercuric iodate crystals

λ(nm)	Absorption coefficient (α)
300	1.226
320	0.993
340	0.736
360	0.508
380	0.325
400	0.206
420	0.17
440	0.139
460	0.12
480	0.086
500	0.08
520	0.089
540	0.07
560	0.071
580	0.063
600	0.055
620	0.068
640	0.052
660	0.052
680	0.051
700	0.057

The band gap energy is determined from absorption spectra with the help of Tauc relation using the recorded excel data. Using this Tauc relation, a graph is plotted between the square root of (αhν) and hν (Photon energy) as shown in the figure 2(a) as well as between the square of (αhν) and hν (Photon energy) as shown in the figure 2(b).

The extrapolation of straight line to $(\alpha h\nu)^2$ axis in figure 2(b) gives the value of Optical band gap energy. The Optical energy band gap of the Mercuric iodate crystals is found to be 1.56 eV. It is in agreement with the value reported elsewhere.

IV. Conclusions

From the above studies we observe that

(I) Gel growth technique is suitable for growing the crystals of Mercuric iodate.
(II) Single diffusion method is convenient for the growth of the Mercuric iodate crystals.
(III) Mercuric iodate crystals of appreciable size can be grown by single diffusion gel technique.
(IV) Gel growth technique is suitable for growing the crystals of mercuric iodate.
(V) The optical band gap energy of the grown crystals is found to be match very well with the reported elsewhere.

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